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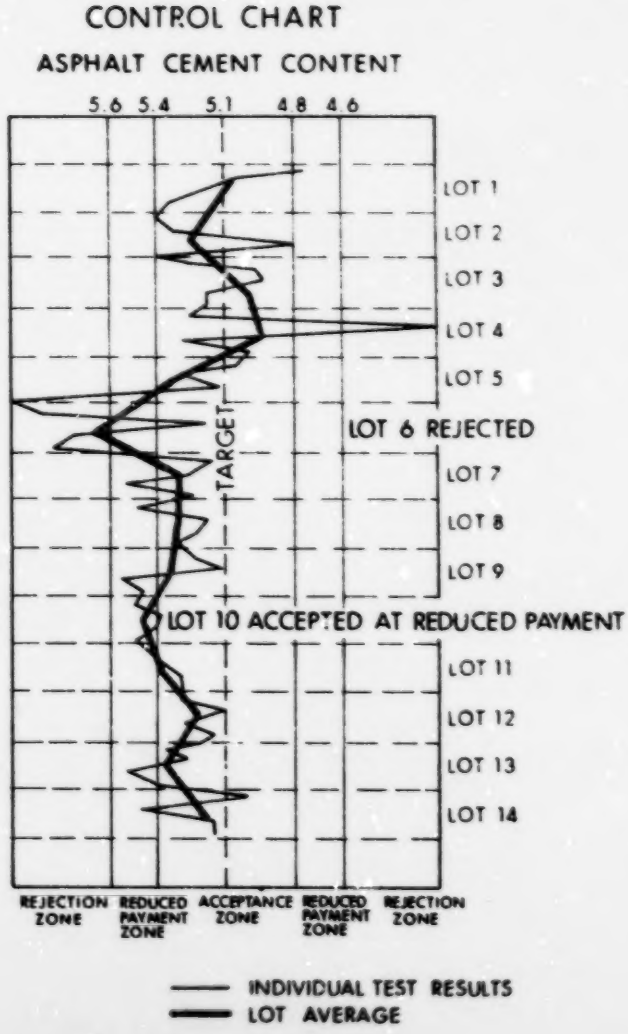
Provincial Highways

Comparison of Methods for the Determination of Bitumen Content in Bituminous Concrete Mixtures



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Bituminous
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by
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Comparison of Methods for the Determination of Bitumen Content in Bituminous Concrete Mixtures

June, 1983

COMPARISON OF METHODS
FOR THE
DETERMINATION OF BITUMEN CONTENT
IN
BITUMINOUS CONCRETE MIXTURES

by

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BITUMINOUS SECTION

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1.0 SUMMARY

This report is both in-house evaluation and literature search of methods used to determine the asphalt content (and in some methods, the aggregate composition) of bituminous concrete mixtures. With the trend to *"rapid on site"* quality control of bituminous concrete and other construction materials, the personnel concerned with Quality assurance should be aware of the advantages and disadvantages of all methods available for the Quality control process.

The writer recommends the use of the more rapid methods for immediate on site control but the user must be aware of the accuracy, sensitivity and the completeness of the results. The report also recommends the continued use of *"extraction type"* processes for payment or quantity reconciliation and mixture aggregate control.

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3.0 RATIONALE

This Ministry presently uses the Rotarex method of determining bitumen content; however, it is known that there are certain errors inherent in this or any of the five other processes.[6] As well, control of bituminous mixes demands both a rapid method of determination and sound statistical quality control. By presenting the facts on these six methods, one will be in a better position to choose the appropriate method(s) to fit their needs.

4.0 METHOD

A review of A.S.T.M. Publication STP 461 *"Rapid Test Methods for Determination of Bitumen Content in Bituminous Mixtures"* (4) along with N.C.H.R.P. #36 *"Instrumentation and Equipment for Testing Highway Materials, Products and Performance"* (5) prompted the purchase and in-laboratory evaluation of the vacuum extractor. It was also felt that the previous experience with the centrifuge (Rotarex), Faultwetter (Reflux) and Troxler Model 2226 nuclear asphalt content gauge should also be reviewed and reported at this same time. With the aid of the *"Computer Assisted Reference Service"* in the MTC Library, a literature search yielded further data and experience by others with these various methods. This search also led us to investigate the *"Pycnometer method"* of determining A.C. content. In 1982 a version of the *"Pycnometer method"* known as the *"Nebraska method"* was also investigated.

Essentially there are two categories of test namely (A) Extraction type, where a solvent is used to dissolve the asphalt cement from the bituminous mixture and (B) Non-Extraction types, such as the nuclear and pycnometer (relative density) methods. These two categories will be discussed separately.

5.0 EXTRACTOR TYPE METHODS

5.1 General

Four of the methods (there are others) of extraction of asphalt cement by solvent are reviewed. The difference between these four methods is purely the physical way the process is carried out and the solvent used.

5.2 Extractors

5.2.1 Rotarex Extractor (A)

The rotarex extractor utilizes centrifugal force to spin off the solvent (trichloroethane) and dissolved bitumen from the sample. In doing so, some fines are lost through the clamped filter. The extract is salvaged and ASTM insists on making a correction for loss of fines. (Our Ministry prefers a total correction factor).

Since this method has been in use for many years by MTC we know and have modified procedures to increase its accuracy and eliminate errors. (See Quality Control of Bituminous Mixes by P. Ksenych, MTC January 1972[6].

5.2.2 Vacuum Extractor (B)

The vacuum extractor utilizes a vacuum to speed up the drawing of extract through the sample and filter. Again as in the previously described method the asphalt is drawn through the filter however with different solvent (dichloromethane). A general correction is suggested by the manufacturer to compensate for both the loss of fines and also the asphalt retained in the sample.

Brakey[3] reported on this extractor in 1965, his findings were confirmed in our laboratory; however, we have had problems with some plant checks giving slow vacuum times. It would appear that this can be remedied by decanting the solvent and flushing the material back into another apparatus.

A drawback in the vacuum process is the low boiling point of the dichloromethane (methylene chloride) which should be stored at room temperature (20°C) or less and thus might lead to potential problems in the field. The process also requires the use of denatured ethyl alcohol which is a highly flammable liquid.

5.2.3 Reflux Extractor (C)

The reflux extractor utilizes the movement of solvent (1,1,1 trichloroethane) by vaporization and gravity. The solvent is placed and boiled in the bottom of the apparatus, the vapours are cooled and condensed at the top of the apparatus and dripped down through the samples, in a continuous process. The use of 20% by volume of denatured alcohol is said to aid the process.

Our experience with a small (1000 g) unit yielded extraction times of from 6 to 13 hours with an average of 8-1/2 hours. The operator is not tied up by the process during this time and can be doing other things. The larger (4000 g) unit will handle a larger sample in shorter times 4 - 6 hours.

5.2.4 Nebraska Pycnometer Method (D)

The Nebraska method is the Pycnometer method with the use of a solvent (1,1,1 trichloroethane) rather than water, the end result being that the asphalt sample is broken down by the solvent and can be dried and graded. Problems with this method are similar to the Pycnometer method which is described in Section 6.2 of this report. The time to yield an asphalt content is 15-20 minutes after the sample has cooled to 80°-90°F. It was also found that not enough of the bitumen is removed to give a satisfactory sample for drying and grading (agglomerations still present).

5.3 Factors Associated With The Extraction Processes

Tables 1 and 2 indicate some of the differences in the four extraction processes.

TABLE 1 - COMPARISON OF EXTRACTORS - PROCESS

Process	Sample Mass (g)	Time of Process	Man Hours	Solvent Used	Remarks
A Rotarex	900	4 h**	4*	trichloroethane	(1) subject to errors such as operator, equipment, speed and fines (2) more fumes from oven drying (3) single correction factor applicable
B Vacuum	1000 - 4000	1 h**	1	dichloromethane (plus alcohol)	(1) sometimes necessary to use diatomaceous earth for filtering (2) less solvent remaining therefore quicker drying and less fumes (3) single correction factor (4) slightly more solvent than in Process A, more than C (5) Solvents must be stored in a cool place
C Reflux	2 @ 500 or 2 @ 2000	8 h**	1	trichloroethane (plus alcohol)	(1) requires cooling water (2) slower than other processes (3) fines from upper sample subject to retention in lower (4) single correction factor (may not be necessary) (5) less solvent used than other processes (6) it is noted that 1400 g may be a practical sample size in each cone in the larger model
D Nebraska	1000 ± 0.1	1-2 h	2	trichloroethane	(1) subject to errors namely differences in B.R.D. of coarse and fine aggregates, B.R.D. of Trichloroethane, absorption of aggregates (2) fumes from oven drying (3) gradation not as effective as the other method (4) excessive fumes for the operator

* During this time several samples are in various stages of the process and thus actual man hours per sample would be in the order of 1 hour/sample.

** Time of process for a complete answer i.e. extraction, drying and grading.

TABLE 2 - COMPARISON OF EXTRACTORS - COST

Type	Model	Cap	Approximate Cost	Cost/Extraction @ $11.00/man h
A	Rotarex D - 1097	1000 g	$1,100.00	44.00
B	Vacuum AP 520	4000 g	$1,800.00	11.00 *
C	AP 183 Reflux	1000 g	$ 800.00	88.00 *
	AP 410	4000 g	$1,025.00	
D	Simple lab apparatus	1000 g	$ 500.00	$11-22

* Operator would be performing more than one check in a laboratory concerned with many contracts so this figure would be less in most instances.

5.4 Correction Factor for Extractors

In the method for the vacuum extractor a process of calibration is used to correct for various gains and losses. This same process can be used for the other extraction processes. In our recent work, it was found that a correction factor for a typical limestone aggregate-bituminous mix is usually positive for the Rotarex method. However, prior to this work a fines correction (centrifuging of fines from extract) had been applied which was a negative correction. Thus it would appear that the centrifuging or ashing correction as suggested by ASTM Method D 2172-81 would normally lead to less accuracy.

The following procedure is recommended to correct for equipment, fines, retained asphalt and operator errors:

The laboratory responsible for the bituminous mix design will, after obtaining an approved mix formulation, prepare two samples of the design mix for each laboratory that will be doing the subsequent mix quality control. These samples are for asphalt extraction purposes and each one will weigh approximately 950 grams. Each laboratory and their operators, will do extraction testing on the duplicate samples received and determine the average asphalt content for the two samples. The two determinations should be within 0.20 of each other, if they are not, a third reference sample shall be run. For a specific laboratory-operator, the difference between the determined asphalt content and the actual-set asphalt content will be the correction factor for that laboratory-operator and that specific mix.

Notes:

1. *The laboratory making up the sets of mixes will prepare them in a "buttered" pan rather than a clean pan unless the pan is sent to the other laboratories along with the mix. The mix may also be shipped in a loose state in a suitable plastic bag, or in a lightly compacted state in a suitable container.*

2. *Correction factors should be established for each contract mix type and for each laboratory involved in the quality control.*

3. *Laboratories involved will in all probability have different factors.*

4. *Each laboratory when reporting results should report both uncorrected and corrected values.*

5. *If correction factors are greater than 0.35 percent, investigate the reasons for the differences and make the necessary changes to the equipment to improve the results. This correction procedure now forms part of NTC Test Method LS-282.*

6.0 NON-EXTRACTOR ASPHALT CONTENT METHODS

6.1 Nuclear Asphalt Content Gauge (E)

Rather than detailing this process, the author would like to make reference to Klotz[1] for an explanation of the principles used in these gauges.

These devices have been around for over two decades, and have been in limited practical use for at least half that time, [4]. The province of Alberta uses these devices for asphalt mix control. Our own experience with the Troxler Model 2226 was reported on in September and November of 1971 by Field in an unpublished report. At that time the gauge showed promise. Drawbacks noted were that the equipment must be calibrated for each aggregate source and the aggregates should be of equal bulk relative density. Hughes[4] noted *"Based on a very limited number of field tests, the nuclear method appears to yield higher values than does the reflux method"*. Field compared his nuclear results with Rotarex (corrected and uncorrected for fines loss) in an on site situation. In general his nuclear values where equal to or lower than the corrected Rotarex values. Steele and Hudson[4] reported a negative bias of 1.08 percent for the nuclear gauge.

Klotz[1] gives a formula for the number of tests (where one test consists of an 11.5 minute counting period) to yield a precision of ± 0.075% A.C. @ 6% Asphalt with 95% confidence.

$n = (1.96\sigma / \pm t)^2$ where t = desired accuracy for overall content determination

σ = mean test method standard deviation

n = number of tests

If n = 4 then it would take approximately 45 minutes for an A.C. Content Determination.

If n = 1 then one would have an asphalt content in about 12 minutes. Field suggested a three point calibration, at design

and plus and minus one percent of the design asphalt content. Klotz[1] suggests that better correlation was found using the two point, zero and design calibration curve. Klotz[1] also suggests that slag aggregates or any aggregates with varying trace iron, boron, manganese and cadmium can vary the absorption microscopic cross-section enough to affect the gauge calibration significantly.

A shorter count can be used with less precision available.

The method does not give one a check on the aggregate gradation. Gradation checks would thus have to be made from the cold feed belts on the asphalt plant.

6.2 Pycnometer Method (F)

This method takes advantage of the following *"when the average relative density (B.R.D.) of the aggregate remains constant, the major factor affecting the maximum relative density (M.R.D.) of the paving mixture is the volume of asphalt cement. Under these conditions the vacuum saturation procedure (James M. Rice) affords a rapid means for determining asphalt content"* [2]. There are two parts to this procedure. The first is to find the maximum relative density (M.R.D.) of the asphalt-aggregate mixture. The second is to determine the ratio of asphalt to total mix that corresponds to this maximum relative density.

$$p = \frac{100\ G_b}{G_a - G_b} \left(\frac{G_a}{G_b} - 1 \right)$$

G_a - effective relative density of the combined mineral aggregate at test temperature.

G_b - relative density of the asphalt at test temperature.

p - percentage of asphalt of total mix by weight.

The effective relative density G_a of the combined aggregates is not necessarily the relative density as determined by standard methods. It is found experimentally by testing specimens of known asphalt content and substituting the known values of P, G_b and G_m in the following equation:

$$G_a = \frac{G_m (100-P)}{100 - G_m P/G_b}$$

G_m - relative density of the mixture at test temperature.

This effective relative density should be determined on ten samples of known asphalt content. Calculate the average of the ten determinations and then determine the maximum plus and minus values from this average. If the values vary more than ± 0.010 discard those values and recalculate the average of the remainder and redetermine the variation. A minimum of six values must be used for the final average.

This method has been found[4] to be more accurate than the reflux method, more precise (for mixtures containing limestone aggregates and for highly absorptive aggregates where extraction methods have problems), however, for slag and gravel aggregates the effective relative density is less consistent and thus the asphalt cement contents are less precise but "still acceptable". For a ± 0.010 variation in the effective relative density, the asphalt content will vary ± 0.1 percent. No expenditure is necessary for solvents and the asphalt content may be determined in about one half to three quarters of an hour. The price of this apparatus would be less than conventional extractors and the apparatus is already available in some regional mobile laboratories where maximum relative densities are already being checked.

As with the nuclear asphalt content gauge, no check on the aggregate gradation can be made by this method. (The Nebraska variation to this method being an exception).

6.3 Factors associated with non extraction process. Table 3 indicates some of the differences of two non extractor processes.

TABLE 3 - COMPARISON OF NON-EXTRACTOR METHODS

Process	Sample Mass (g)	Time of Process	Man Hours	Cost/Determination @ $11.00/man h	Approximate Cost of Apparatus
E Nuclear	6000	15-45 min	1/2-1	6 - 11.00	$11,000.
F Pycnometer	1000 -2000	30-60 min	1/2-1	6 - 11.00	$ 500.

7.0 SURVEY OF OTHER ROAD AGENCIES

In an internal report by G. Cautillo and H. Rait concerning Statistical Quality Assurance, some twenty-four agencies plus MTC Ontario were contacted on the use of the various processes. From this report the following summary is presented:

Out of 25 agencies, four do not use an extraction process, two of these control the mix by bulk relative density of the mix, one controls by tank dipping (material control), and one uses the nuclear asphalt content gauge. Of the twenty-one that use extractions only ten identified the process used. Four used the rotarex, four the reflux, one the vacuum extractor and one the Nebraska pycnometer method.

8.0 SUMMARY AND RECOMMENDATIONS

The vacuum extractor is not recommended unless adequate cool storage is available for the dichloromethane and the extract.

The reflux extractor is slower than the rotarex but where an adequate source of cooling water is available this method can easily be used for a statistically based control of asphalt content and aggregate grading.

The cost of the nuclear asphalt content gauges is prohibitive. The process does not give a picture of the aggregate grading and would require the sampling of the aggregate cold feed which has its own problems.

The rotarex method is presently used by our Ministry. It gives reasonable answers in a reasonable length of time and should be continued to be used.

The Nebraska (pycnometer method) has drawbacks which would hamper its accuracy if put into full scale use, however in some situations would be very quick and accurate.

The extraction methods give the full story on the bituminous mixture provided an appropriate correction factor is applied. However, if these relatively slower processes were augmented by the Pycnometer method, the additional check on asphalt content would be available for early use on the job site.

9.0 ACKNOWLEDGEMENTS

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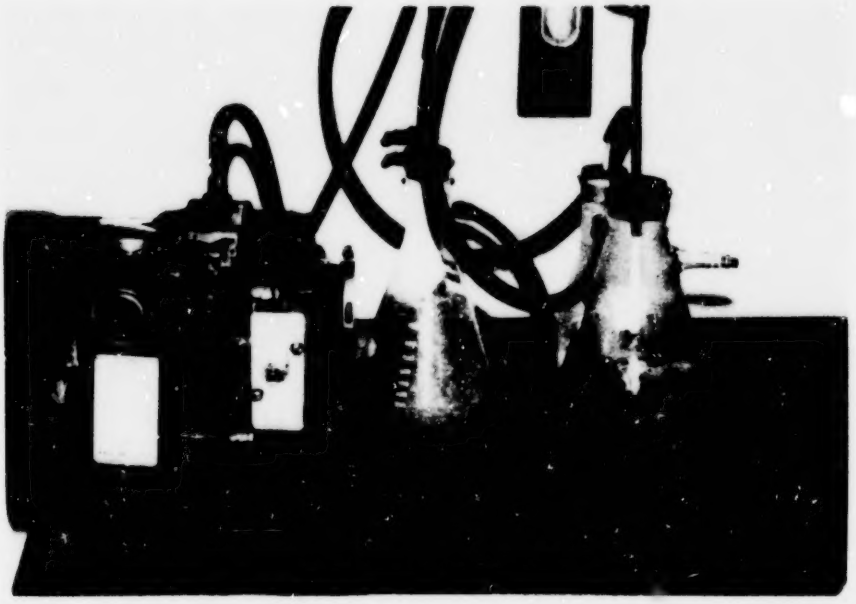
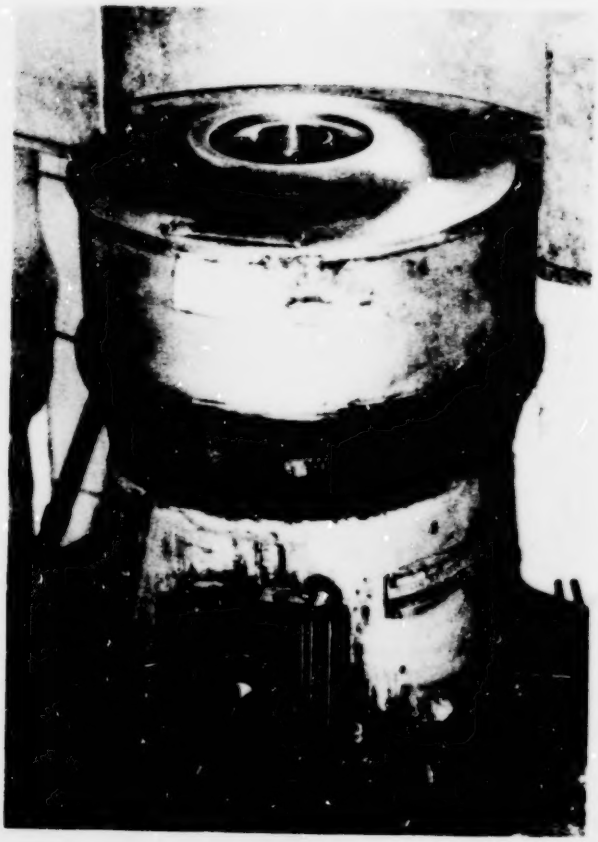
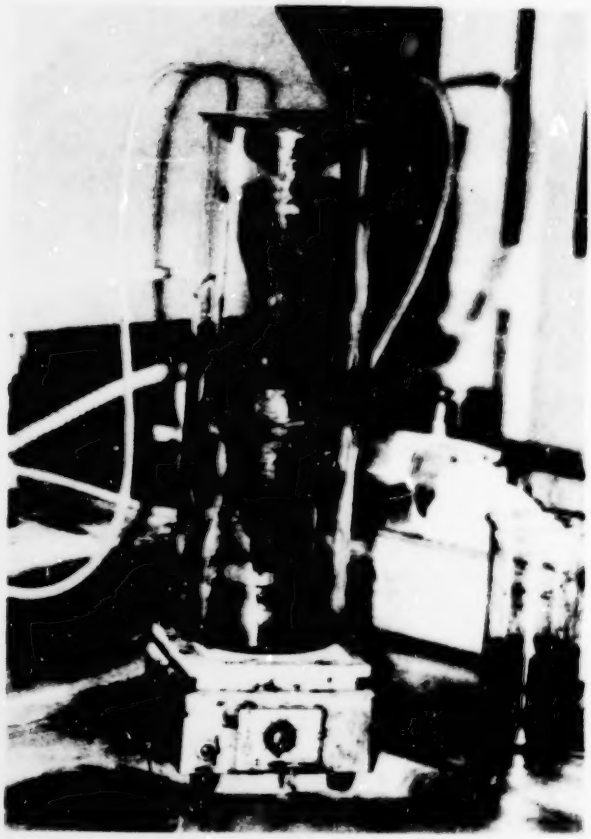


Pycnometer Method - Vacuum Saturation

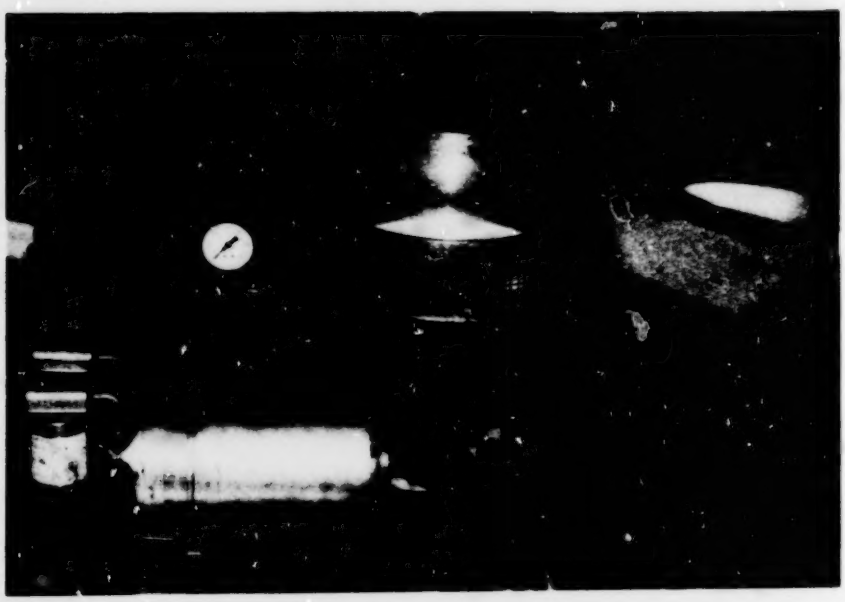
Vacummed Sample being weighed



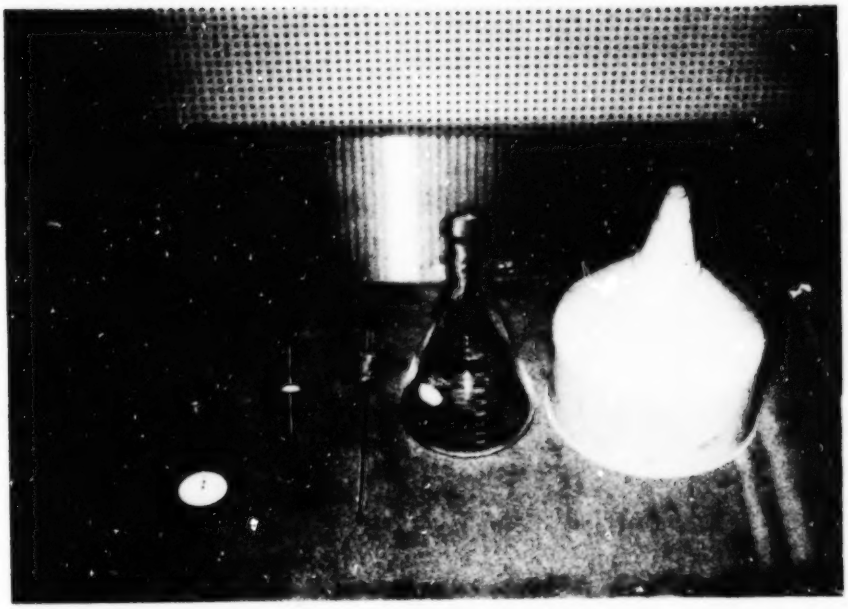
Dulin Rotarex

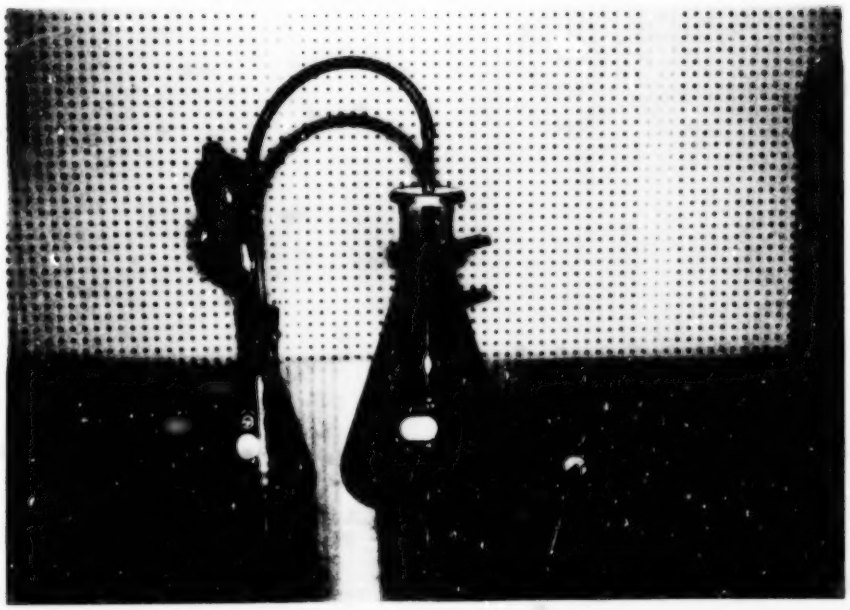


Reflux



Vacuum Extractor





Nebraska Method

11.0 REFERENCES

1. Klotz, Robert C., Penn. D.O.T. - *"Asphalt Content Determination Using Nuclear Techniques"*, HRR #468, 1973, pp: 115-129.

2. Kandahl, Prithvi S., Koehler, William C. and Wenger, Monroe E., Penn. D.O.T. - *"Rapid Determination of Asphalt Content Using Pennsylvania Pycnometer"*, HRR #468, 1973, pp: 89-99.

3. Brakey, B. A., Colorado D.O.H. - *"A Vacuum Extractor for Bituminous Mixtures"*, Materials Research and Standards, December 1965.

4. ASTM STP 461 - *"Rapid Test Methods for Determination of Bitumen Content in Bituminous Mixtures"*, June 1968.

5. NCHRP #36 - *"Instrumentation and Equipment for Testing Highway Materials, Products and Performance"*.

6. Ksenych, P. - *"The Quality Control of Bituminous Concrete Mix Production"*, January 1972.

7. Texas Highway Department - *"Determination of Asphalt and Moisture Content by Nuclear Method"*, 1971 - 08 - 00.

8. Farr, C. R., Millions, K. A., Anderson, K. O. - *"Asphalt Content Determination Techniques on Conventional and Dryer-Drum Mixed Asphaltic Concrete"* - Proceedings of 21st Annual Conference, Canadian Technical Asphalt Association, November 1976.

9. Troxler Laboratores Inc., - *"Asphalt Content Gauge"* Model 2226, P.O. Box 5997, Raleigh, North Carolina 27607.